Refine Search

Search Results -

Terms	Documents
L9 and butylhypochlorite	0

Database: US Pre-Grant Publication Full-Text Database; US Patents Full-Text Database; US OCR Full-Text Database; EPO Abstracts Database; JPO Abstracts Database; Derwent World Patents Index; IBM Technical Disclosure Bulletins

Search: L11

Refine Search | Recall Text | Clear | Interrupt

Search History

DATE: Monday, March 06, 2006 Printable Copy Create Case

Set Name side by side	Query	Hit Count	Set Name result set
DB=USPT,USOC,EPAB,JPAB,DWPI,TDBD; PLUR=YES; OP=ADJ			
L11	L9 and butylhypochlorite	0	L11
L10	L9 and (560/$ or 568/$)	6	L10
L9	L7 and ((tin or nickel) or (sn or ni))	39	L9
L8	L7 and (tin or nickel) or (sn or ni)	976542	L8
L7	L6 and reduc$7	55	L7
L6	L5 and phosgene	62	L6
L5	L4 or l3	119	L5
L4	phenylisocyanate and nitrotoluene	51	L4
L3	phenyl isocyanate and nitrotoluene	88	L3
L2	phneylisocyanate and nitrotoluene	0	L2
DB=PGPB; PLUR=YES; OP=ADJ			
L1	20040147776	1	L1

END OF SEARCH HISTORY

Hit List

First Hit | Clear | Generate Collection | Print | Fwd Refs | Bkwd Refs | Generate OACS

Search Results - Record(s) 1 through 6 of 6 returned.

☐ 1. Document ID: US 6503949 B1

Using default format because multiple data bases are involved.

L10: Entry 1 of 6 File: USPT Jan 7, 2003

US-PAT-NO: 6503949
DOCUMENT-IDENTIFIER: US 6503949 B1

TITLE: Glucagon antagonists/inverse agonists

DATE-ISSUED: January 7, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Lau; Jesper	Farum			DK
Madsen; Peter	Bagsv.ae butted.rd			DK
Sams; Christian	Frederiksberg C			DK
Behrens; Carsten	Copenhagen N			DK
Vagner; Josef	Oro Valley	AZ		
Christensen; Inge Th.o slashed.ger	Lyngby			DK
Lundt; Behrend Frederik	Kokkedal			DK
Sidelmann; Ulla Grove	Vedb.ae butted.ek			DK
Th.o slashed.gersen; Henning	Farum			DK
Ling; Anthony L.	San Diego	CA		
Plewe; Michael Bruno	San Diego	CA		
Truesdale; Larry Kenneth	San Diego	CA		
Shi; Shenghua	San Diego	CA		

US-CL-CURRENT: 514/617; 514/613, 560/105, 560/12, 560/129, 560/51, 560/8, 562/433, 562/455

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw De

☐ 2. Document ID: US 4946854 A

L10: Entry 2 of 6 File: USPT Aug 7, 1990

US-PAT-NO: 4946854
DOCUMENT-IDENTIFIER: US 4946854 A

TITLE: 3-aminobenzoylphenylureas useful for controlling parasites and insects that

attack domestic animals and livestock

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw De

☐ 3. Document ID: US 4740610 A

L10: Entry 3 of 6 File: USPT Apr 26, 1988

US-PAT-NO: 4740610
DOCUMENT-IDENTIFIER: US 4740610 A
** See image for Certificate of Correction **

TITLE: Process for the preparation of N,o-substituted mono- and/or polyurethanes

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw De

☐ 4. Document ID: US 4695645 A

L10: Entry 4 of 6 File: USPT Sep 22, 1987

US-PAT-NO: 4695645
DOCUMENT-IDENTIFIER: US 4695645 A

TITLE: Process for the preparation of N,o-substituted mono-and/or polyurethanes

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw De

☐ 5. Document ID: US 4547322 A

L10: Entry 5 of 6 File: USPT Oct 15, 1985

US-PAT-NO: 4547322
DOCUMENT-IDENTIFIER: US 4547322 A

TITLE: Method for manufacture of diphenylmethane diisocyanates

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw De

☐ 6. Document ID: US 4290970 A

L10: Entry 6 of 6 File: USPT Sep 22, 1981

US-PAT-NO: 4290970
DOCUMENT-IDENTIFIER: US 4290970 A

TITLE: Process for the manufacture of aromatic di- and/or polyisocyanates

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw De

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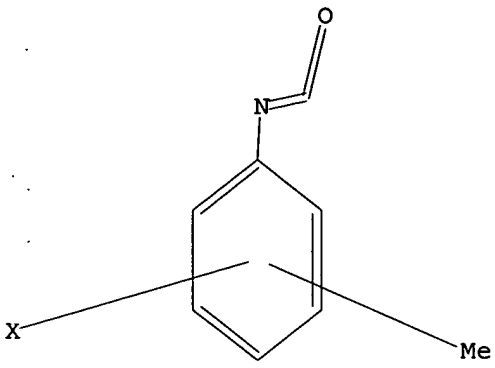
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               S L1

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     FILE 'CAPLUS' ENTERED AT 17:22:36 ON 06 MAR 2006
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L4            22 S L3 AND NITROTOLUENE
L5             0 S L3 AND BUTYLHYPOCHLORITE
L6            10 S L4 AND PY<2003
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L1 STRUCTURE UPLOADED

=> d
L1 HAS NO ANSWERS
L1 STR



Structure attributes must be viewed using STN Express query preparation.

=> s l1 full
REGISTRY INITIATED
Substance data SEARCH and crossover from CAS REGISTRY in progress...
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

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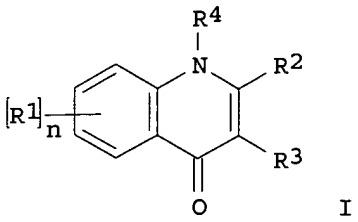
L6 ANSWER 1 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2004:36703 CAPLUS
DOCUMENT NUMBER: 140:93939
TITLE: Preparation of 1,2-disubstituted 1,4-dihydro-4-oxoquinoline compounds as antiviral agents
INVENTOR(S): Tamura, Takashi; Kuriyama, Haruo; Agoh, Masanobu; Agoh, Yumi; Soga, Manabu; Mori, Teruyo
PATENT ASSIGNEE(S): Maruishi Pharmaceutical Co., Ltd., Japan
SOURCE: Eur. Pat. Appl., 44 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1380575	A1	20040114	EP 2003-18235	20000829
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL				
JP 2001064259	A2	20010313	JP 1999-242700	19990830 <--
JP 3521264	B2	20040419		
JP 2001064261	A2	20010313	JP 1999-242701	19990830 <--
JP 3259089	B2	20020218		
JP 2001089455	A2	20010403	JP 1999-262883	19990917 <--
JP 2001089476	A2	20010403	JP 1999-262884	19990917 <--
EP 1081138	A1	20010307	EP 2000-118673	20000829 <--
EP 1081138	B1	20040922		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
AT 277017	E	20041015	AT 2000-118673	20000829
ES 2228370	T3	20050416	ES 2000-118673	20000829
US 2004009977	A1	20040115	US 2003-369578	20030221

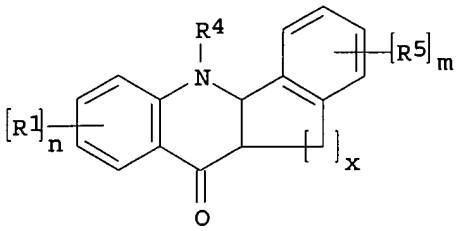
PRIORITY APPLN. INFO.:

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JP 1999-262884	A	19990917
EP 2000-118673	A3	20000829
US 2000-649596	A3	20000829

OTHER SOURCE(S): MARPAT 140:93939
GI



I



II

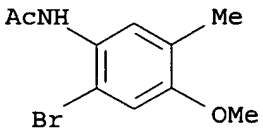
AB The title compds. [I; R1 = alkyl, cycloalkyl, Ph, etc.; n = 1-3; R2 = alkyl, pyridyl, furyl, etc.; R3 = H, alkyl, Ph, etc.; if R2 = Ph optionally substituted with halo or alkoxy, R3 may represent a bridging group between the 3rd position of the quinoline ring and said Ph at a position next to the ring carbon atom at which said Ph is directly connected to the quinoline ring, said bridging being selected from CH2, CO, 1,2-ethylidene, etc.; if R2 = 2-thienyl, 5-alkyl-2-thienyl or N-alkylpyrrol-3-yl, R3 may represent CH2; R4 = alkyl, alkenyl, CH2Ph, Ph] having antiviral activity, were prepared When the bridge is formed of a C1-3 alkylidene, the compound of the invention has formula II [R1, R4, n as

above; x = 1-3; R5 = H, halo, alkyl, alkoxy; m = 1-2]. Thus, treating 1-indanone with n-BuLi and tetramethylenediamine in THF followed by addition of 1-ethyl-6-isopropylisatoic anhydride (preparation given) afforded II [x = 1; R1 = 8-iso-Pr; R4 = Et; R5 = H]. The compds. I were tested in vitro for anti-picornavirus activity and for anti-rhinovirus activity (data given).

IT **645419-16-5P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of 1,2-disubstituted 1,4-dihydro-4-oxoquinoline compds. as antiviral agents)

RN 645419-16-5 CAPLUS

CN Acetamide, N-(2-bromo-4-methoxy-5-methylphenyl)- (9CI) (CA INDEX NAME)



REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

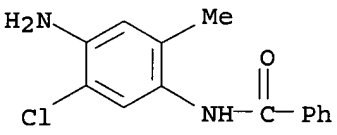
L6 ANSWER 2 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1953:23816 CAPLUS
DOCUMENT NUMBER: 47:23816
ORIGINAL REFERENCE NO.: 47:4085e-g
TITLE: Naphthol dyes. I. Synthesis of Fast Violet B base
AUTHOR(S): Konishi, Kenzo; Nishiura, Ayaru
CORPORATE SOURCE: Naniwa Univ., Sakai
SOURCE: J. Soc. Org. Synthet. Chem. (Japan) (**1952**), 10, 542-5
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB 2-Amino-4-chlorotoluene was condensed in Na2CO3 with p-toluenesulfonyl chloride at 95° to 2-(p-tolylsulfonamido)4-chlorotoluene, further nitrated with 15% HNO3 at 80° to 2-p-tolylsulfonamido-4-chloro-5-**nitrotoluene**, deacylated with 80% H2SO4 at 110-15° to 2-amino-4-chloro-5-**nitrotoluene** (I), methoxylated with MeOH-KOH at 130° to 2-amino-4-methoxy-5-**nitrotoluene**, benzoylated in toluene by boiling with BzCl over an oil bath to 2-benzamido-4-methoxy-5-**nitrotoluene**, and finally reduced by boiling over a water bath with Fe dust (in EtOH) and 5% HCl to 2-benzamido-4-methoxy-5-aminotoluene (Fast Violet B base), yield above 80%; with Raney Ni catalyst above 90%. From I by similar benzoylation and reduction was obtained 2-benzamido-4-chloro-5-aminotoluene which gave fast brown with naphthol dyes.

IT **812665-69-3**, o-Benzotoluidide, 4'-amino-5'-chloro-
(preparation of)

RN 812665-69-3 CAPLUS

CN o-Benzotoluidide, 4'-amino-5'-chloro- (5CI) (CA INDEX NAME)



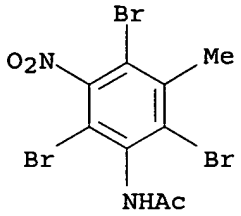
L6 ANSWER 3 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1947:11816 CAPLUS
DOCUMENT NUMBER: 41:11816
ORIGINAL REFERENCE NO.: 41:2397h-i,2398a-h
TITLE: Qualitative separation of the isomeric nuclear-substituted tetrabromonitrotoluenes
AUTHOR(S): Qvist, Walter

CORPORATE SOURCE: Abo Akad., Abo, Finland
SOURCE: Acta Acad. Aboensis, Math. et Phys. (**1940**), Volume Date 1939, 12(No. 1), 17 pp.
DOCUMENT TYPE: Journal
LANGUAGE: German

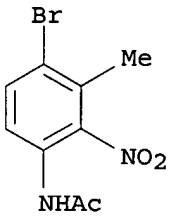
AB In a study of the composition of the nitration products of tetrabromcymene other than 2,3,5,6-tetrabromo-4-**nitrotoluene** (I), the isomers of I were prepared and studied. p-MeC6H4NH2 (25 g.) in 2 l. 10% HCl was treated with 75 g. Br. The solid material, filtered, washed with H2O, and recrystd. from EtOH, gave 53 g. 3,5-dibromo-4-aminotoluene (II), m. 73-4°. II (60 g.), treated according to Nevile and Winther (Ber. 13, 974(1880)), washed with EtOH and Et2O, and recrystd. from EtOH, yielded 40 g. 3,4,5-tribromotoluene (III), m. 90.5-1.5°. III (20 g.), 2 g. iodine, 2 g. Fe, and 3.2 ml. Br in 200 g. CCl4 were heated at 50° in daylight until all the Br had reacted (a few days to 3 weeks, depending upon the season). The reaction product was washed with Na2S2O3 solution, with H2O, dried, and evaporated The residue, recrystd. from EtOH, yielded 19.8 g. 2,3,4,5-tetrabromotoluene (IV), m. 111.5-12.5°. IV (2 g.) was gently heated with 40 ml. fuming HNO3 until solution was complete (0.5 hr.). Cooling yielded 1.5 g. crude 3,4,5,6-tetrabromo-2-**nitrotoluene** (V) which, boiled with an equal amount of piperidine in EtOH and then recrystd. from EtOH, yielded pure V, m. 220.5-1.5°. Longer heating with fuming HNO3 or heating with HNO3 and H2SO4 resulted in lower yields. In a similar manner, starting with m-MeC6H4NH2, were prepared 2,4,6-tribromo-3-aminotoluene (VI), m. 102.5-3°, and 2,3,4,6-tetrabromotoluene (VII), m. 112-12.5°. VII (2.5 g.) and 75 ml. fuming HNO3 were kept at room temperature several hrs. and then slowly heated until solution was complete. Cooling yielded 1.95 g. crude crystals which, purified with EtOH and piperidine, yielded 2,4,5,6-tetrabromo-3-**nitrotoluene** (VIII), m. 224.5-5.5°. Nitration of VII with concentrated H2SO4 and fuming HNO3 gave demethylated and other by-products. VI (5 g.), 1.55 g. Ac2O, a few drops H2SO4, and some AcOH heated on the H2O bath until solution was complete, and the resulting product recrystd. from dilute EtOH gave 2,4,6-tribromo-3-acetamidotoluene (IX), m. 211.5-12°. IX gradually added to cold fuming HNO3 and then poured onto ice gave 2,4,6-tribromo-5-nitro-3-acetamidotoluene (X), m. 269-9.5° (EtOH). X was heated with concentrated H2SO4 at 110° 10 min., cooled, and poured onto ice. The precipitate, washed and recrystd. from EtOH, gave 2,4,6-tribromo-5-nitro-3-aminotoluene (XI), m. 188.5-9.5°. XI (10 g.) in cold concentrated H2SO4 was treated with nitrous gases, diluted with H2O, and treated with KBr and Br. The precipitated perbromide was filtered, carefully washed with H2O, EtOH, and Et2O, and decomposed by solution in warm AcOH. The resulting precipitate (7.2 g.), purified by means of piperidine and EtOH and recrystd. from EtOH, yielded 4.7 g. VIII. Nitration of tetrabromocymene, purification with piperidine and EtOH, and recrystn. from EtOH yielded I, m. 217.5-18.5°. Mixed m.ps. of various percentages of I and V, I and VIII, and V and VIII showed no depression. Reduction of I, V, and VIII with Zn and AcOH at room temperature gave solid products which were easily identified. V (100 mg.) was treated with 0.5 g. Zn and 10 ml. AcOH and allowed to stand 24 hrs. at room temperature The mixture was diluted with H2O, filtered, and extracted with Et2O. The Et2O extract was evaporated and the residue, dissolved in EtOH, precipitated with H2O, and recrystd. from 50% EtOH, yielded 3,5,6-tribromo-2-aminotoluene (XII), m. 85-5.5°. XII was diazotized with nitrous gases in dilute HNO3, diluted with H2O, and heated with EtOH a few hrs. on the H2O bath. The precipitate, filtered and crystallized from dilute MeOH, gave 2,3,5-tribromotoluene (XIII), m. 52-2.5°, did not depress the m.p. of authentic XIII. Nitration of XIII with fuming HNO3 and concentrated H2SO4 yielded 3,5,6-tribromo-2,4-dinitrotoluene, m. 213.5-14.5°. Similar reduction of VIII and recrystn. from EtOH yielded 2,4,6-tribromo-3-aminotoluene (XIV), m. 102.5-3°; mixed m.p. with XII, 77-80°. Removal of the NH2 group as above gave 2,4,6-tribromotoluene (XV), m. 68-9°. Similar reduction of I and recrystn. from 30% EtOH gave 2,6-dibromo-4-aminotoluene, m. 89-90°, which upon bromination yielded XV. XIII was prepared by brominating o-MeC6H4NH2 in HCl solution to form 3,5-dibromo-2-aminotoluene, m. 47-8°, which was deaminized by diazotization as described above. XV was prepared similarly from m-MeC6H4NH2.

IT **857569-22-3**, m-Acetotoluidide, 2',4',6'-tribromo-5'-nitro-

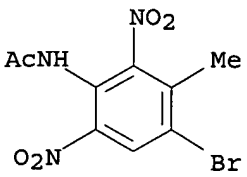
(preparation of)
RN 857569-22-3 CAPLUS
CN m-Acetotoluidide, 2',4',6'-tribromo-5'-nitro- (5CI) (CA INDEX NAME)



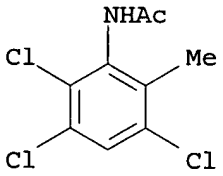
L6 ANSWER 4 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1945:5940 CAPLUS
DOCUMENT NUMBER: 39:5940
ORIGINAL REFERENCE NO.: 39:918i,919a-b
TITLE: Nitro amino derivatives of o-bromobenzoic acid
AUTHOR(S): Goldstein, Henri; Preitner, Gaston
SOURCE: Helvetica Chimica Acta (**1944**), 27, 888-91
CODEN: HCACAV; ISSN: 0018-019X
DOCUMENT TYPE: Journal
LANGUAGE: French
AB Nitration of 0.52 g. of 2,5-Br(AcNH)C6H3CO2H (prepared by a slight modification of the method of Bamberger, C. A. 19, 1258) with 0.8 cc. HNO3 (d. 1.50) at room temperature for 20 min. gave 0.50 g. (83%) of 5-acetamido-2-bromo-6-nitrobenzoic acid (I), m. 112°, identical with the product obtained by the oxidation of 1.08 g. of 5-acetamido-2-bromo-6-**nitrotoluene** (II) with 2 g. KMnO4 in 50 cc. H2O in the presence of 2 g. MgSO4 in 99 cc. H2O by refluxing for 3-4 h. and crystallizing the product from AcOH. I was saponified to the corresponding 5-amino-2-bromo-6-nitrobenzoic acid, m. 218°. II was obtained in the nitration of 11.5 g. of 5-acetamido-2-bromotoluene, m. 103-4°, which yielded 7 g. of 5-acetamido-2-bromo-4-**nitrotoluene** (III), m. 126-7°, II and some 5-amino-2-bromo-6-**nitrotoluene**, m. 103°, and a small amount of 5-acetamido-2-bromo-4,6-dinitrotoluene, m. 224-5°. Oxidation of III produced the corresponding 5-acetamido-2-bromo-4-nitrobenzoic acid, m. 208°, saponified to 5-amino-2-bromo-4-nitrobenzoic acid, m. 236.5°, in 77% yields.
IT **857617-22-2**, m-Acetotoluide, 4-bromo-2-nitro- **857617-25-5**, m-Acetotoluide, 4-bromo-2,6-dinitro-
(preparation of)
RN 857617-22-2 CAPLUS
CN m-Acetotoluide, 4-bromo-2-nitro- (4CI) (CA INDEX NAME)



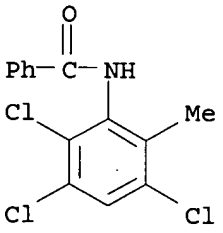
RN 857617-25-5 CAPLUS
CN m-Acetotoluide, 4-bromo-2,6-dinitro- (4CI) (CA INDEX NAME)



L6 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1931:13802 CAPLUS
DOCUMENT NUMBER: 25:13802
ORIGINAL REFERENCE NO.: 25:1501d-e
TITLE: Chlorination of o- and p-**nitrotoluene**. 3,4,6-Trichloro-2-**nitrotoluene** and 2,6-dichloro-4-**nitrotoluene**
AUTHOR(S): Levy, Leopold F.; Stephen, Henry
SOURCE: Journal of the Chemical Society (**1931**) 76-9
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB o-O2NC6H4Me (27.4 g.) and 5 g. SbCl5, saturated with Cl, give 5 g. of the 3,4,6-cl3 derivative, pale yellow, m. 93-4°; further nitration gives the 2,5-di-NO2 derivative, m. 229.5° (corrected). Reduction with SnCl2 and HCl gives 3,4,6-trichloro-o-toluidine, m. 89°; Ac derivative, m. 199°; Bz derivative, m. 230°. p-O2NC6H4Me, chlorinated as above, gives the 2,6-Cl2 derivative, m. 63-4°; HNO3 and H2SO4 give the 3,4-di-NO2 derivative, m. 130°. 2,6-Dichloro-p-toluidine, m. 55°; Ac derivative, m. 215°; Bz derivative, m. 179°. Both of the new Cl derivs. resist oxidation after heating with KMnO4 for 6 days on the water bath.
IT **860580-73-0**, o-Acetotoluide, 3,5,6-trichloro- **861078-70-8**, o-Benzotoluide, 3',5',6'-trichloro-
(preparation of)
RN 860580-73-0 CAPLUS
CN o-Acetotoluide, 3,5,6-trichloro- (3CI) (CA INDEX NAME)



RN 861078-70-8 CAPLUS
CN o-Benzotoluide, 3',5',6'-trichloro- (3CI) (CA INDEX NAME)



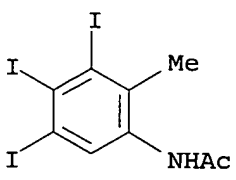
L6 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1927:557 CAPLUS
DOCUMENT NUMBER: 21:557
ORIGINAL REFERENCE NO.: 21:90d-i,91a-c
TITLE: Substituted indole-2-carboxylic-3-propionic acids and some iodinated benzene derivatives
AUTHOR(S): Kalb, Ludwig; Schweizer, Franz; Zellner, Heinrich; Berthold, Erich
SOURCE: Ber. (**1926**), 59B, 1860-70
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB The object of the present work was to synthesize, by the method described in the preceding abstract, substituted indole-3-propionic acids, especially I derivs. in connection with the problem of thyroxin, whose constitution, however, has now been cleared up by Harington (C. A. 20, 2506). Et 1-4'-nitrobenzeneazo-2-cyclopentanone-1-carboxylate, red, m. 50°,

converted by boiling 1 hr. with 30% alc. H_2SO_4 into di-Et α-ketoadipate p-nitrophenylhydrazone (yield, 90%), brown-yellow, m. 163°, while 50% H_2SO_4 gives 78% di-Et 6-nitroindole-2-carboxylate-3-propionate, m. 163°; free di-CO_2H acid (yield, 86%), yellowish, m. 268-70° (decomposition). p-Iodophenylhydrazone of α-ketoadipic acid (I) (yield, 65%), yellowish, m. 169°, converted by boiling 4 hrs. with 20% alc. H_2SO_4 into 5-iodoindole-2-carboxylic-3-propionic acid (56% yield), m. 252° (decomposition), which is also obtained in 65% yield from indole-2-carboxylic-3-propionic acid in AcOH with ICl. Et 1-3',4',5'-triiodobenzeneazo-2-cyclopentanone-1-carboxylate, m. 191° (decomposition) in sealed capillaries, insol. in cold dilute NaOH, soluble on boiling with transient green, then yellow color and formation of the 3,4,5-triiodophenylhydrazone of I (95% yield), m. 197° (decomposition) in a sealed tube, 3 g. of which on gentle boiling for 4 hrs. with 30% alc. H_2SO_4 gives 2 g. 4,5,6-triiodoindole-2-carboxylic-3-propionic acid, yellowish, m. 248° (decomposition) on rapid heating in a sealed tube. Et 1-3',5'-diiodo-4'-methoxybenzeneazo-2-cyclopentanone-1-carboxylate (yield, 81%), light yellow, m. 173°, gives with NaOH on the H_2O bath 88% of the 3,5-diiodo-4-methoxyphenylhydrazone of I m. 192° (decomposition), easily soluble in dilute Na_2CO_3, which with 30% alc. H_2SO_4 yields 86% of di-Et 4,6-diiodo-5-methoxyindole-2-carboxylate-3-propionate, m. 163°; di-CO_2H acid (yield, 78%), m. 236-8° (decomposition). 2,6,4-$I_2(O_2N)C_6H_2NH_2$, obtained in 95% yield from 50 g. p-$O_2NC_5H_4NH_2$ with 150 g. ICl in AcOH at 60°, can be diazotized only in concentrated H_2SO_4 and yields with KI 90% of 3,4,5-$I_2C_8H_3NO_3$, m. 167°, which is reduced in boiling Me_2CO by $SnCl_2$-HCl to 3,4,5-$I_3C_6H_2NH_2$ (yield, 95%), m. 174.5° (decomposition), unchanged by several days' exposure to direct July sunlight; this can be diazotized only in concentrated HNO_3 and seps. as the very difficultly soluble diasonium nitrate, yellow, only little sensitive to moderate heat but detonates over the free flame, can be dried in a desiccator but slowly decomps.; attempted diazotization of the amine in concentrated HCl suspension with solid $NaNO_2$ gave the diazoamino compound, m. 250° (decomposition). 2,5-$O_2N(H_2N)C_6H_3Me$ with 2 mols. ICl gives 59% 4-iodo-2-nitro-5-aminotoluene-HCl (free base, yellow, m. 177-8°) and 29% 4,6-diiodo-2-nitro-5-amino-toluene, m. 146-6°, which in 70% H_2SO_4 with solid $NaNO_2$ and then with KI gives 4,5,6-triiodo-2-**nitrotoluene** (yield, 70%), yellowish white, m. 139.5°, reduced in AcOH by boiling $SnCl_2$-HCl to the 2-amino compound (yield, 85%), light yellow, m. 132-3° (Ac derivative, m. 269.5°). 2,5-$O_2N(H_2N)C_6H_3CO_2H$, obtained in 56% yield from 20 g. $O_2N(AcNH)C_6H_3Me$ and 44 g. $MgSO_4$ in boiling H_2O with 60 g. $KMnO_4$, gives on deacetylation and treatment in dilute Na_2CO_3 with excess of ICl at room temperature 4,6-diiodo-2-nitro-5-aminobenzoic acid, golden yellow, m. 276°, which on diazotization in concentrated H_2SO_4 and treatment with KI yields 4,5,6-triiodo-2-nitrobenzoic acid, yellow, m. 235°. Treatment of p-$O_2NC_6H_4OH$ in 10% NH_4OH with I and decomposition of the resulting di-NH_4 salt with HCl yields 2,6,4-$I_2(O_2N)C_6H_2OH$, which, dissolved in hot Me_2SO_4 and slowly treated with 20% NaOH, yields 94.5% of the Me ether, brownish, finely granular powder, m. 134-5°; this in hot alc. with $SnCl_2$-HCl gives 94% 2,6-diiodo-4-aminoanisole, m. 100°; HCl salt, m. 214° (decomposition), hydrolyzed in H_2O to the base; sulfate, m. 186-7°; Ac derivative, m. 200°. 2,6,4-$I_2(H_2N)C_6H_2OH$-HCl, obtained in 90% yield from the NO_2 compound in boiling alc. with $SnCl_2$-HCl, m. 218-20° (decomposition) (sulfate, m. 192-3° (decomposition)); on attempted diazotization in alc. HCl it yields 2,6-diiodo-1-quinone 4-diazide, light brown, deflagrates on heating, does not couple.

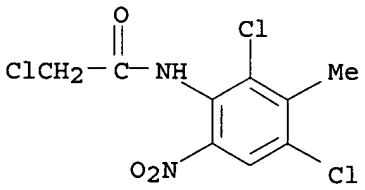
IT **859945-81-6**, o-Acetotoluide, 3,4,5-triiodo-
(preparation of)

RN 859945-81-6 CAPLUS

CN o-Acetotoluide, 3,4,5-triiodo- (3CI) (CA INDEX NAME)



L6 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1926:22935 CAPLUS
DOCUMENT NUMBER: 20:22935
ORIGINAL REFERENCE NO.: 20:2833h-i,2834a-b
TITLE: Study of the rule of the conservation of the type of substitution in aromatic substances. I. Chlorination of 2-chloro-4-**nitrotoluene**
AUTHOR(S): Davies, Wm.; Leeper, G. W.
SOURCE: Journal of the Chemical Society (**1926**) 1413-9
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB cf. C. A. 16, 806. D. found that in the chlorination of 2,4-Cl(O2N)C6H3Me in the presence of certain catalysts the yield of the 2,6-Cl2 derivative (I) is 30 % and that no other isomer could be isolated. Holleman (C. A. 18; 3175) points out that other isomers might be present but not isolated. Further study now shows that this is the principal Cl derivative, the reaction product containing considerable amts. of substances other than nuclear-substituted dichloro-4-nitrotoluenes; the yield of I was raised to 47.9%. This result, when compared with the nitration of 2,4-Cl(O2N)C6H2Me, in which more than 85% of the product is the 4,5-di-NO2 derivative, shows that a change of substitution type has occurred. Nitration of I gives 99.8% of 2,6,3,4-Cl2(O2N)2C6HMe (II), m. 130°; reduction gives an oxidizable o-diamine, characterized as 1,3-dichloro-2-methylbenzophenanthrazine, yellowish brown, m. 279-86° (corrected); its formation from II is not quant. and the process does not lend itself to a rough estimation of II in the presence of other o-di-NO2 derivs. of PhMe. Reduction of II with EtOH-NH3 gives 77% of 2,6-dichloro-4-nitro-m-toluidine, deep yellow (C6H6), brown (EtOH), m. 136°; Ac derivative, m. 185°; Bz derivative, m. 215°; chloroacetyl derivative, m. 170°. The determination of I as II is described, as is the effort to isolate other products from the mother liquor of I. Further chlorination of I gives a mixture of products, from which no definite product was isolated; the portion m. 60-78° on nitration and reduction gives with phenanthraquinone a compound C21H11N2Cl3, deep yellow, m. 343°, which may be 1,3,4-trichloro-2-methylbenzophenanthrazine.
IT **861343-27-3**, m-Acetotoluide, α,2,4-trichloro-6-nitro- (preparation of)
RN 861343-27-3 CAPLUS
CN m-Acetotoluide, α,2,4-trichloro-6-nitro- (2CI) (CA INDEX NAME)



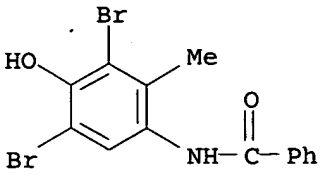
L6 ANSWER 8 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1912:2245 CAPLUS
DOCUMENT NUMBER: 6:2245
ORIGINAL REFERENCE NO.: 6:354i,355a-i
TITLE: Chloroiminoquinones
AUTHOR(S): Raiford, L. C.
SOURCE: American Chemical Journal (**1912**), 46, 417-56
CODEN: ACJOAZ; ISSN: 0096-4085
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB 2-Bromo-4-nitrophenol (a) was prepared from its Ba salt by treatment with HCl, colorless needles from H2O, m. 112°. 2-Bromo-4-aminophenol hydrochloride (b) was prepare by the action of concentrate HCl upon (a) in presence of SnCl2. Yellowish crystals decompose without m. 225°. By treatment of (b) with (NH4)2CO2, 2-bromo-4-aminophenol, brownish needles

from C6H6, m. 165°, is formed. At 0°, 4-chloroimino-2-bromoquinone (I), O: C4H3Br: NCl, is formed by treatment of (b) with an HCl solution of NaClO, crystals from ligroin, decompose without m. 60°. 4-Chloroimino-2-chloro-6-bromoquinone (II), O : C6H2BrCl: : NCl was prepared as (I) from the phenol, m. 137°, and the hydrochloride decompose 225°, crystals from ligroin, m. 87-8°, decompose 175°. 2,4,6-Trichloro-3-methylphenyl acetate, C9H7O2Cl3, separated as an oil, distilling at 273-4° from the action of Ac2O in presence of Na acetate, upon trichloro-m-cresol, 2,6-dichlorotoluquinone (c) was prepared by oxidizing the m-cresol. Yield, 72% crystals from ligroin and alc., m. 103°. A saturated solution of SO2, acting upon (c) produced 2,6-dichlorotoluhydroquinone, colorless needles, m. 171°. On nitration with NaNO2 of tribromo-m-cresol, a mixture of isomeric mononitrodibromo-m-cresols, m. 113°, separated as a yellow solid. The p-compound (d) 2,6-dibromo-4-nitro-m-cresol precipitated from ligroin, m. 134°. 0-Compound (e) 2,4-dibromo-6-nitro-m-cresol was obtained on evaporate of ligroin filtrate and crystalline from alc., m. 87°. The aminocresol hydrochloride was obtained from (d) as (b), decompose 225°. The aminocresol of (d) from alc., m. 176°, 2,6-dibromo-4-benzoylamino-m-cresol, C14H11O2NBr2, was obtained by treating the aminocresol with BzCl, crystals from CHCl3 and ligroin, m. 198°. 3,5-Dibromo-6-methyl-p-hydroxyphenylurethan, HOC6HBr2MeNHCO2Et, was obtained by treating the aminocresol of (d) with EtCO2Cl and HCl. Crystals from CHCl3 and ligroin, m. 155°. 4-Chloramino-2,6-dibromotoluquinone (III), O : C6HBr2Me : NCl, was obtained as (I), m. 86°. The aminocresol hydrochloride of (e), prepared as (b) from (e) was less soluble in H2O than p-compound The aminocresol of (e) m. 116-7°. The benzoate of (e) was prepare as benzoate of (d), m. 188°. Urethan of (e) prepare as urethan of (d), yield 60%. Crystals, m. 169°. The aminophenyl acetate of (e) from cresol of (e). Colorless, silky needles from alc., m. 216°. 2,4-Dibromo-3-methyl-6-nitrophenylethyl carbonate, O2NC6HBr2MeOCO2Et, was prepared in presence of NaOH by treatment of (e) with EtCO2Cl. The oil solidified from glacial AcOH, m. 43-5°. By reduction of the carbonate with Sn and HCl, the urethan m. 169° was prepared (IV). Three chloroiminochlorotoluquinones were prepared as (I): 6-Chloroimino-4-chlorotoluquinol, m. 91°, obtained from Kehrmann's chloroaminocresol. 6-chloroimino-2-chlorotoluquinone, O : C6H2ClMe : NCl, m. 87°, obtained from chloroaminocresol; 4-chloromino-2-methyl-5-chloroquinone, m. 65°, obtained from 2-methyl-4-amino-5-chloro-0-cresol. 2-Chloro-4-benzoylamino-5-methylphenyl benzoate was prepared from the aminocresol, m. 206-7°, as benzoate of (d), m. 220°. 3-Chloroimino-4-chlorotoluquinone, O : C6H2ClMe : NCl, was obtained as (I) from 3-nitro-4-chlorotoluene, crystals from ligroin, m. 65°. 2-Chloro-6-nitro-m-cresol, HO.C6H2ClMeNO2, was obtained by chlorinating 6-nitro-m-cresol, crystals from benzene, m. 133°. Hydrochloride of (f) prepare as (b), m. 250°. Aminocresol of (f) prepare as (c), m. 166-7°. The acetate of (f) m. 178°. 2-Chlorotoluhydroquinone (V), O : C6H2ClMe; O, was obtained by treatment of aminocresol of (f) with cold dichromate mixture. Crystals, m. 55°. 2-Chlorotoluhydroquinone, HOC6H2ClMeOH, was obtained by treatment of (V) with saturated solution of SO2, colorless leaflets from H2O, m. 173°. On passing Cl (1 mol.) into dry 2-**nitrotoluene** a mixture of isomers 6-chloro-2-nitrotolueue and 4-chloro-2-**nitrotoluene** b. 236-7° are formed. The mixture was reduced by Sn and HCl to the toluidine and by glacial AcOH to the acetotoluide. On crystallization 6-chloro-2-nitroacetotoluidine was formed, m. 154°, and 4-chloro-2-nitroacetotoluidino, m. 136-7°. The mixture of the nitrotoluenes by elec. reduction and rearrangement in H2SO4 produced the corresponding pair of phenols: 1-Methyl-2-chloro-6-aminophenol and 1-methyl-4-chloro-6-aminophenol which in turn, by oxidation, give p-chlorotoluquinone, m. 105° and 0-chlorotoluquinone, m. 55°.

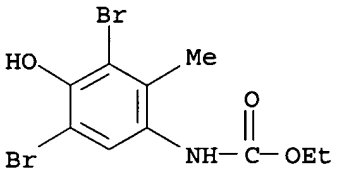
IT **861308-06-7**, o-Benzotoluide, 3',5'-dibromo-4'-hydroxy- (and derivs.)

RN 861308-06-7 CAPLUS

CN o-Benzotoluide, 3',5'-dibromo-4'-hydroxy- (2CI) (CA INDEX NAME)



IT **861579-12-6**, Carbanilic acid, 3,5-dibromo-4-hydroxy-2-methyl-, ethyl ester
(preparation of)
RN 861579-12-6 CAPLUS
CN Carbanilic acid, 3,5-dibromo-4-hydroxy-2-methyl-, ethyl ester (1CI) (CA INDEX NAME)



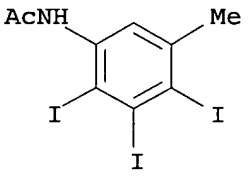
L6 ANSWER 9 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1911:2678 CAPLUS
DOCUMENT NUMBER: 5:2678
ORIGINAL REFERENCE NO.: 5:476g-i,477a-d
TITLE: Iodine Derivatives of Toluene
AUTHOR(S): Wheeler, Henry L.
CORPORATE SOURCE: Sheffield Lab., Yale Univ.
SOURCE: American Chemical Journal (**1911**), 44, 493-507
CODEN: ACJOAZ; ISSN: 0096-4085
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB The remainder of the 19 possible nuclear-substituted I derivs. of MePh are described. 3-Nitro-o-toluidine (22.5 g.) and ICl (24.5 g.) in glacial AcOH (20 g.) give a 98% yield of 5-iodo-3-nitro-o-toluidine, orange needles from alc., m. 135°. When diazotized and treated with KI this gives an 81% yield of 2,5-diiodo-3-**nitrotoluene**; colorless prisms from alc., m. 95°, on reduction it gives 2,5-diiodo-m-toluidine, pale brown prisms from alc., m. 82°, yield 60%. 2,5-Diiodo-m-acetotoluide, from the base and Al2O, long, colorless, felt-like needles from alc., m. 198-9°. On warming 7 g. 2,5-diiodo-m-toluidine, I (5 g.), CaCO3 (4 g.), Et2O (20 cc.), and H2O (15 cc.) on the steam bath for 18 hrs., a 50% yield of 2,5,6-triiodo-m-toluidine, colorless needles from alc., m. 119-120°, was obtained; with 4 ats. I, 2,4,5,6-tetraiodo-m-toluidine (see below) was obtained. On diazotization and treatment with KI, the triiodo derivative gave a 64% yield of 2,3,5,6-tetraiodotoluene, colorless arborescent needles, from C6H6, m. 125°. 4,5-Diiodo-o-toluene from I or ICl and p-iodo-o-toluidine, stout needles or long prisms from alc., m. 85°, it yielded 3,4,6-triiodotoluene on diazotization and treatment with KI. p-Nitro-o-toluidine and ICl, in glacial AcOH, gave a 70% yield of 4-nitro-5-iodo-o-toluidine, long, bright yellow prisms from alc., m. 109°. From this was prepared 2,5-diiodo-4-**nitrotoluene**, buff-colored prismatic plates from alc., m. 117°. On reduction it gave 2,5-diiodo-p-toluidine, buff-colored prisms from alc., m. 109°, this yielded 2,4,5-triiodotoluene, which was incorrectly called by Neumann, a 2,4,6-derivative (Ann., 240, 50). A 31% yield of 2,4,6-triiodotoluene was obtained by the action of HNO2 on 2,4,6-triiodo-m-toluidine, colorless needles from alc., m. 105°. 5-Nitro-o-toluidine and ICl (1 or 2 mols.) gave a 90% yield of 3-iodo-5-nitro-o-toluidine, long, brown, prisms from alc., m. 173°. When diazotized and treated with KI this gave a 93% yield of 2,3-diiodo-5-**nitrotoluene**, long, light-brown needles from alc., m. 143°. 5,6-Diiodo-m-toluidine, colorless crystals from alc., m. 106°.

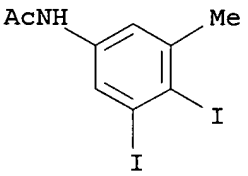
5,6-Diiodo-m-acetotoluide spike-like clusters from alc., m. 208°. On diazotization and treatment with KI, both 2,5-diiodo-m-toluidine and 5,6-diiodo-m-toluidine yield 3,5,6-triiodotoluene, large, light orange, twinned plates from alc., m. 72-3°. 5,6-Diiodo-m-toluidine and I yielded 4,5,6-triiodo-m-toluidine, small brown needles from alc., m. 122°, and 2,4,5,6-tetraiodo-m-toluidine, small needles from glacial AcOH, m. 205°. 4,5,6-Triiodo-m-acetotoluide, from the base and AcCl, colorless needles, m. 265°. 2,3,4-Triiodotoluene, needles from alc., m. 92°, was prepared by the action of HNO2 on 4,5,6-triiodo-m-toluidine, which also gave a 63% yield of 3,4,5,6-tetraiodotoluene, straw-yellow needles from C6H6, m. 284-5°. A 20% yield of pentaidotoluene was obtained by diazotizing the tetraiodotoluidine and treating the solution with KI, dull yellow needles from C6H6 m. about 340° (decompose).

IT **861525-15-7**, m-Acetotoluide, 4,5,6-triiodo- **861611-74-7**, m-Acetotoluide, 4,5-diiodo- **861611-76-9**, m-Acetotoluide, 2,5-diiodo-
(preparation of)

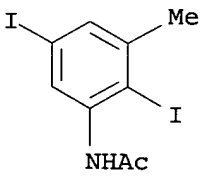
RN 861525-15-7 CAPLUS
CN m-Acetotoluide, 4,5,6-triiodo- (1CI) (CA INDEX NAME)



RN 861611-74-7 CAPLUS
CN m-Acetotoluide, 4,5-diiodo- (1CI) (CA INDEX NAME)



RN 861611-76-9 CAPLUS
CN m-Acetotoluide, 2,5-diiodo- (1CI) (CA INDEX NAME)



L6 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1910:15606 CAPLUS
DOCUMENT NUMBER: 4:15606
ORIGINAL REFERENCE NO.: 4:2796e-i,2797a-d
TITLE: Action of Iodine on Metatoluidine
AUTHOR(S): Wheeler, Henry L.
CORPORATE SOURCE: Sheffield Lab., Yale Univ.
SOURCE: American Chemical Journal (**1910**), 44, 126-45
CODEN: ACJOAZ; ISSN: 0096-4085
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

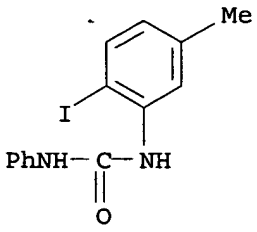
AB When 1 mol. of m-toluidine is mixed with I (2 or 3 ats.) in presence of Et2O, H2O and excess of CaCO3 mono- and diiodotoluidines are formed; using 4 or 6 ats. of I, triiodotoluidines are also formed. 6-Iodo-m-toluidine

is best prepared by the action of 1 mol. ICl on m-acetotoluide dissolved in glacial AcOH, and treating the resulting acetyl compound, m. 132-4° with alc. KOH, yield 74%. The same compound was prepared by 2 other methods; m. 37-9, Artmann (Monatsh., 26, 1099) gives m. p. 98-9°, and acetyl derivative, m. 132°. 6-Iodo-m-acetotoluide is oxidized by KMnO4 to 6-iodo-m-acetaminobenzoic acid, yield 52%. Prismatic needles from hot H2O, m. 210°. Unlike the isomeric iodoaminobenzoic acids, 6-iodo-3-aminobenzoic acid could not be isolated from the acetyl derivative 4,6-Diiodo-m-toluidine from m-toluidine, 6-iodo-m-toluidine or 4-iodo-m-toluidine and I, thick colorless needles from alc., turning brown in air, m. 73-4°. Hydrochloride, long needles from alc. Acetyl derivatives, C7H5I2NHAc, colorless needles, m. 213°, it is very inert towards KMnO4. 2,4,6-Triiodo-m-toluidine, from 4,6-diiodo-m-toluidine or 2,6-diiodo-m-toluidine and 2 ats. of I; pale brown hairlike needles, m. 135°. 2,4,6-Triiodo-m-acetotoluidide, C7H4I3NHAc, needles from alc., m. 265°. By adding a diazo solution of 2,4,6-triiodo-m-toluidine to KI, 2,3,4,6-tetraiodotoluene was formed, long needles from C6H6, m. 170°. 2,6-Diiodo-m-acetotoluide, from 2-iodo-m-acetotoluide and ICl, colorless prisms from alc., m. 171°, yield 78%. This acetyl derivative when saponified by alc. KOH forms 2,6-diiodo-m-toluidine, colorless needles and prisms from alc., m. 88°, yield 82%. Hydrochloride. On treating a diazonium solution of 2,6-diiodo-m-toluidine with KI, 2,3,6-triiodotoluene is formed, colorless needles from alc., m. 80.5°, yield 93%. On reducing 2,6-dinitrotoluene with (NH4)2S, 6-nitro-o-toluidine was obtained, yield 96%. The latter amine and I form 5-iodo-6-nitro-o-toluidine, yellow prisms from alc., m. 85°, yield 70-87%. This base when diazotized and treated with KI forms 2,5-diiodo-6-**nitrotoluene**, colorless needles from alc., m. 105°, yield quantitative. The latter compound on reduction by FeSO4 and NH4OH forms 3,6-diiodo-o-toluidine, colorless needles from alc., m. 86°, yield 67%. When diazotized and treated with KI, this base forms 2,3,6-triiodotoluene. 4-Iodo-3-**nitrotoluene** is reduced with FeSO4 and NH4OH forming 4-iodo-m-toluidine, m. 38-38.5°. Willgerodt and Simonis (Ber., 39, 273) give m. p. 48°. Hydrochloride, m. 155° (Willgerodt and Simonis, 90-150°). Thiocarbamide derivative, C6H3IMeNHCSNHPh, crystals from alc., m. 162-3°. 4-Iodo-m-acetotoluide from the base and Ac2O, m. 151° (Willgerodt and Simonis, 145-6°). With ICl this acetyl derivative forms 4,6-diiodo-m-acetotoluide. 4-Iodo-m-toluidine and I yield 4,6-diiodo-m-toluidine. By diazotizing the latter compound and treating the solution with KI, 3,4,6-triiodotoluene is formed, brownish needles from alc., m. 119-20°, yield 60%. 3-Nitro-p-toluidine and ICl form 3-nitro-5-iodo-p-toluidine, golden brown needles from alc., m. 98°, yield 68%. This base when diazotized and treated with KI forms 3-nitro-4,5-diiodotoluene, orange rectangular prisms, m. 84-5°, yield 64%. When reduced with FeSO4 and NH4OH this yields 4,5-diiodo-m-toluidine, colorless cotton-like needles from dilute alc., m. 66-7%. The base and AcCl yield 4,5-diiodo-m-acetotoluide, colorless needles, m. 183-4°(?) When 5-nitro-m-toluidine is diazotized and treated with KI, 3-nitro-5-iodotoluene is formed, yellow rectangular prisms, m. 77°. This yields on reduction by FeSO4 and NH4OH, 5-iodo-m-toluidine, colorless asbestos-like needles, m. 78-78.5°.

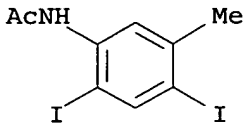
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(preparation of)

RN 860749-99-1 CAPLUS

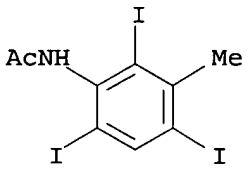
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RN 861525-24-8 CAPLUS
CN m-Acetotoluide, 4,6-diiodo- (1CI) (CA INDEX NAME)



RN 861549-49-7 CAPLUS
CN m-Acetotoluide, 2,4,6-triiodo- (1CI) (CA INDEX NAME)



RN 861611-78-1 CAPLUS
CN m-Acetotoluide, 2,4-diiodo- (1CI) (CA INDEX NAME)

